

**THE SILVER/SILVER SULFIDE REFERENCE
ELECTRODE FOR USE IN CORROSION STUDIES
IN KRAFT WHITE LIQUOR**

Project 3556

**Report One
A Progress Report
to
MEMBERS OF THE INSTITUTE OF PAPER CHEMISTRY**

February 15, 1984

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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SUMMARY

Various electrochemical techniques for assessing corrosion damage in kraft mill liquors involve measurement of the potential that exists at the metal/liquor interface. Every such measurement requires the use of a reference electrode which is electrically connected to the negative (-) terminal of the voltage measuring instrument to provide a voltage reference point. Conventional reference electrodes are not suitable for direct-immersion use in kraft liquors because they are either poisoned by the sulfurous species in the liquors or are damaged by the high temperatures and pressures encountered.

The silver/silver-sulfide (SSS) electrode has been examined and found to be suitable for direct-immersion use as a reference electrode in kraft white liquors. The SSS electrode offers a stable reference potential in various liquors (~ -880 mVSCE), is easily fabricated, and is easily installed and maintained in probes used for corrosion measurements. The reference potential is weakly dependent on the sulfide concentration of the liquor, and a calibration curve of potential vs. $[S^{2-}]$ is provided. Detailed instructions on fabrication and use of the electrode are also provided.

INTRODUCTION

Corrosion and corrosion-assisted cracking of metals in contact with kraft liquors are essentially electrochemical processes involving anodic and cathodic reactions at the metal surface. Because of this electrochemical involvement, the potential difference at the interface between the metal and the liquor controls the rate at which corrosion damage occurs. The potential developed spontaneously at the metal/liquor interface is a good qualitative indicator of the corrosion damage occurring on exposed metal surfaces.

Various electrochemical methods which are available for more exact measurement of corrosion damage rates will also require the measurement of the potential developed at the metal/liquor interface. With these methods, the potential is artificially controlled at some level by passing a controlled current through the interface. The relative magnitudes of the voltage change, $\Delta\epsilon$, and the incremental current required to induce this change, Δi , provide diagnostic information about the corrosion process. In some cases, commercial instrumentation is available to make automatic measurements using these techniques.

Virtually every electrochemical test to assess corrosion behavior will involve measurement of the potential developed at the metal/liquor interface. These measurements can not be made reliably without a trustworthy reference electrode that can be immersed in the liquor and connected to the voltmeter as a voltage benchmark.

The essential feature of a reference electrode is its ability to maintain a stable reference potential charge at its surface when it is immersed in various electrolytes. The voltage measured in any test will include both the voltage change at the reference electrode as well as the metal surface, so

errors in reference potential have direct impact on the accuracy of any potential measurement. It is also important that the reference potential be related to a universal standard for reference potential, such as the 0.000V condition ascribed to the standard hydrogen electrode (SHE).

Many reference electrodes have been identified and used over the years (1), including the familiar silver/silver-chloride electrode (reference potential = 0.224 V vs. SHE) and the saturated Calomel electrode (reference potential = 0.241 V vs. SHE). These reference electrodes maintain a stable potential by exposing a noble metal (e.g., Ag or Hg) and one of its salts (AgCl, Hg₂Cl) in a small compartment containing ions of the metal at known concentration. A known concentration of metal ions in the solution is maintained by a dissociative equilibrium between the metal, the salt, and the anions of the salt in the solution. For example, in the Ag/AgCl electrode shown in Fig. 1, the dissociation constant for AgCl is given in terms of the activities by

$$K_s = a_{\text{Ag}^+} \cdot a_{\text{Cl}^-} \quad (1)$$

The activity of the Cl⁻ ion is controlled by addition of KCl (or some similar soluble chloride salt) to the chamber in known concentration. The activity of silver ions, a_{Ag^+} , is thereafter held at

$$a_{\text{Ag}^+} = \frac{K_s}{a_{\text{Cl}^-}} \quad (2)$$

The potential at the surface of contact between the Ag metal and the solution containing Ag⁺ ions is established by the Nernst Equation.

$$E = E_{\text{Ag/Ag}^+}^{\circ} + \frac{RT}{F} \ln(a_{\text{Ag}^+}) \quad (3)$$

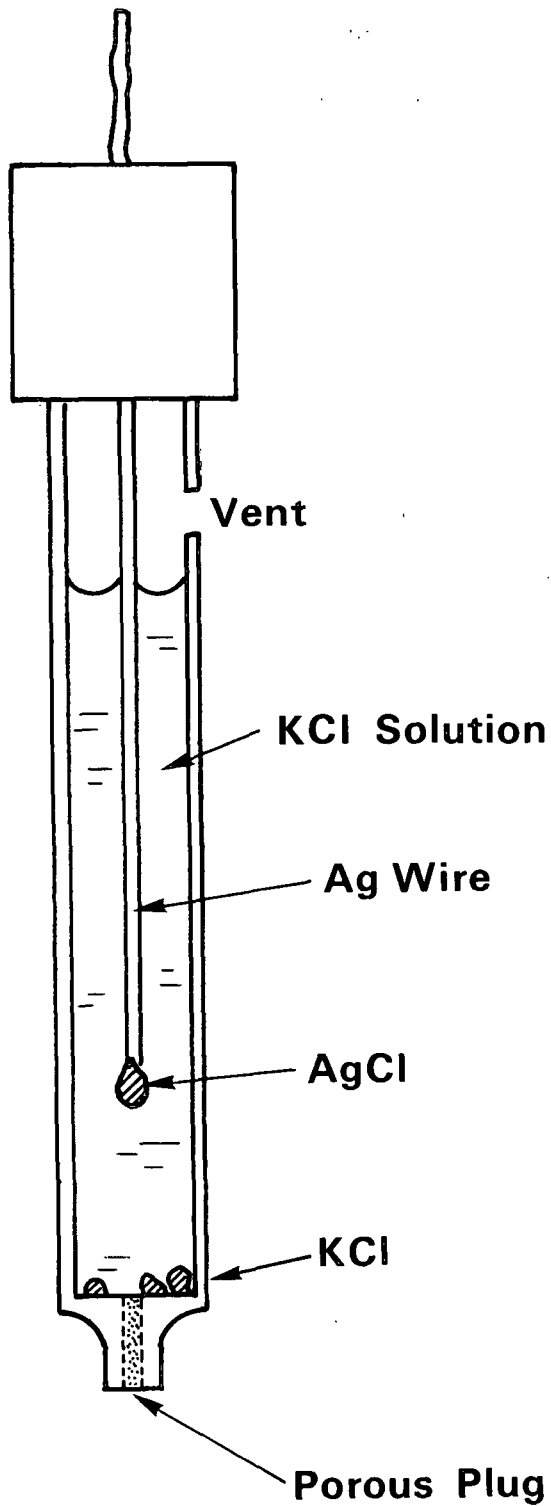


Figure 1. Schematic diagram of the Ag/AgCl reference electrode.

where R = gas constant

T = absolute temperature

F = Faraday's constant

and E_{Ag/Ag^+}^0 = standard reduction potential for $Ag/Ag^+ = 0.2224$ V vs. SHE

Eq. (3) may be written as

$$\begin{aligned} E &= E_{Ag/Ag^+}^0 + \frac{RT}{F} \ln \frac{K_s}{a_{Cl^-}} \\ &= 0.799 \text{ V} + \frac{RT}{F} \ln \frac{K}{a_{Cl^-}} \end{aligned} \quad (4)$$

Substituting for K_s , and setting $T = 298^\circ K$,

$$E_{Ag/AgCl} = 0.2224 - 0.059 \log a_{Cl^-} \quad (5)$$

When the concentration of KCl in the compartment is 1 molar,

$$E_{Ag/AgCl} = 0.2224 \text{ V vs. SHE}$$

Electrolytic contact between the electrolyte in the compartment and the liquor is maintained through a porous plug in the housing of the reference electrode body. Obviously, care must be taken to prevent contamination of the KCl electrolyte in the inner compartment to maintain a stable potential.

Although there are many reference electrodes for use in laboratory settings, few of these electrodes are durable enough for use in field testing. Most commercially available reference electrodes are made of glass which is not chemically resistant or rugged enough for field service. The problem with reference electrode durability is particularly acute in kraft liquors because of reactions with the sulfur compounds present in these liquors. For example, the Calomel

electrode immersed in kraft liquors soon becomes inoperative because of precipitation of mercurous sulfide in the porous plug junction between liquor and the electrode compartment. A final drawback to laboratory-type reference electrodes is that they are incompatible with the screw-on probes (see Fig. 2) typically used in routine corrosion testing because they need a liquid-filled KCl compartment.

Several approaches have been used to circumvent the problems associated with the use of generally available reference electrodes in kraft liquors. In some cases, a noble metal (molybdenum, platinum, stainless steel) is used as a reference electrode but the potential of such electrodes is very sensitive to temperature and the concentration of oxidizing species in the liquor. Furthermore, it is virtually impossible to relate the reference potential of such electrodes to any generally accepted standard value. Others have used laboratory-type reference electrodes, but have isolated the electrodes by means of a salt-bridge as shown in Fig. 3. The salt bridge transmits the potential to the reference electrode via a long, high conductivity electrolytic path between the reference electrode and the kraft liquor. Typically, such a salt bridge might be a Teflon tube encasing an asbestos wick saturated with KCl solution. This approach circumvents problems with temperature and pressure damage and with electrode poisoning by sulfur compounds. However, a complicated experimental arrangement with fragile reference electrodes is still required in this approach, and the apparatus remains incompatible with typical corrosion sensor hardware.

Attention was focused on the silver/silver sulfide electrode as a candidate for direct immersion use in kraft liquor. This electrode was attractive

because it appeared that it could be immersed directly into the kraft liquor without an intermediate plenum of a controlled-composition liquid. The dissolved sulfides in kraft liquors would act as the Cl^- acts in the Ag/AgCl electrode to control the concentration of Ag^+ . If this electrode could maintain a stable potential, it would be very attractive as a reference electrode because of its durability, ease of fabrication, and use and compatibility with existing corrosion sensor probes.

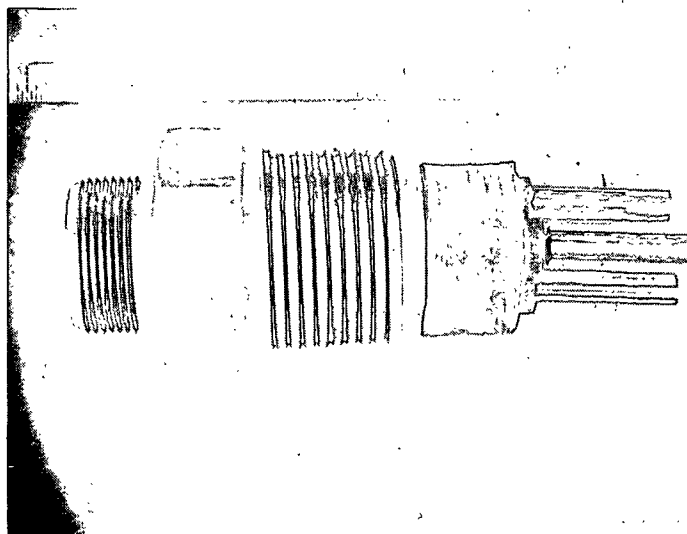


Figure 2. A typical three-electrode probe used for corrosion testing in the field.

To establish the feasibility of using the SSS electrode as a reference electrode, tests were conducted (1) to establish the rest potential in several simulated and actual liquors of various compositions and temperatures, (2) to characterize the reproducibility of the reference potential and the extent of fluctuations during prolonged exposures, (3) to document reference potential stability in the presence of organic species and inorganic oxidants, and (4) to

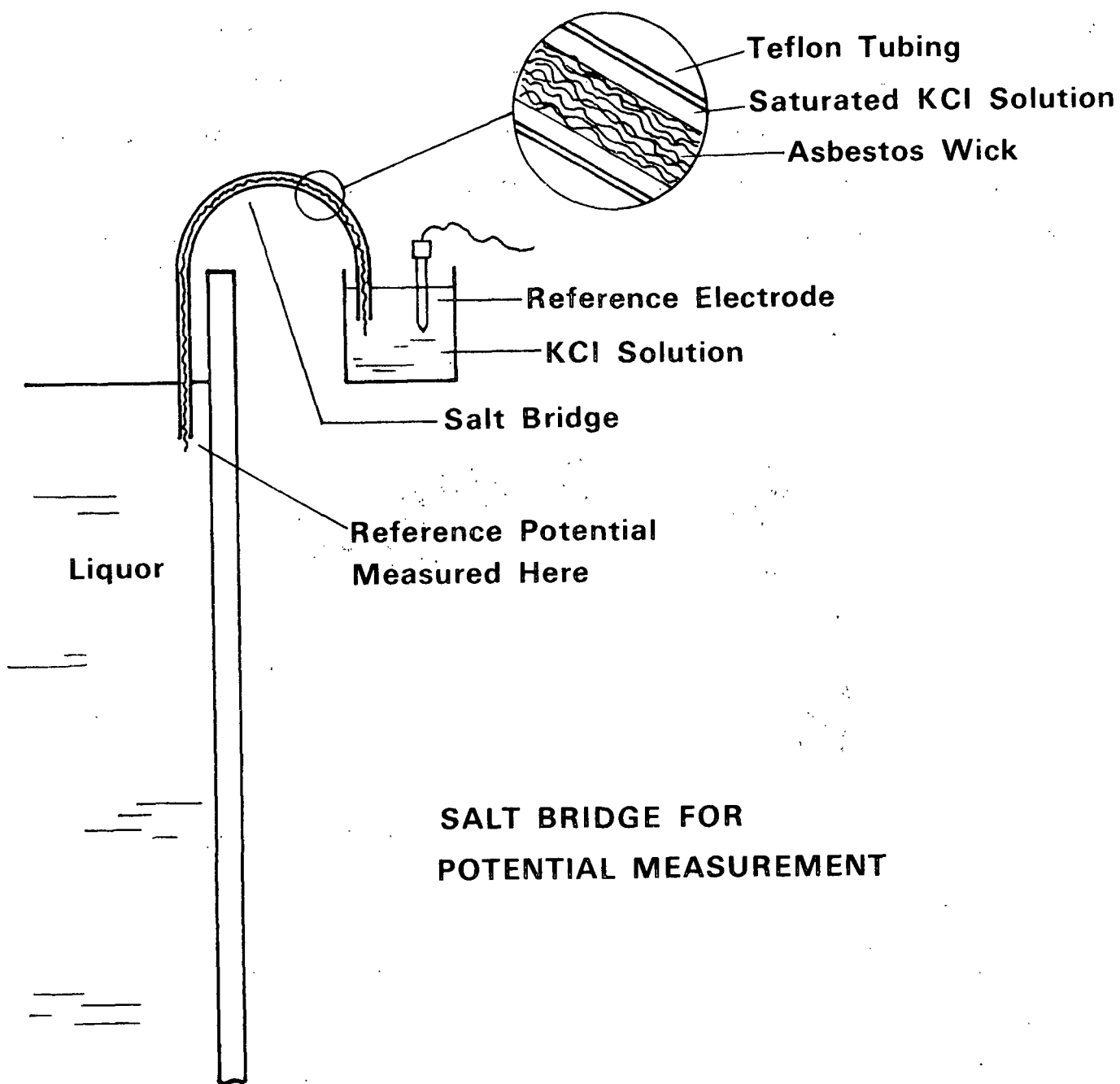


Figure 3. Use of a salt bridge to prevent damage to fragile reference electrodes during potential measurements in kraft liquors.

assess the feasibility of the electrode for use in field studies. In most cases, the investigation involved comparisons of the SSS electrode potential with the potential of a Calomel electrode that was physically isolated but electrolytically in contact with the liquors.

EXPERIMENTAL PROCEDURE

ELECTRODE PREPARATION

SSS electrodes were prepared by anodizing internally threaded silver rods, 6 mm o.d. x 15 mm, in an alkaline sulfide solution to form a surface film of silver sulfide. Silver rods were purchased from Materials Research Corporation, Bridgeton, MO; the cost of silver for each electrode was ~ \$15. Electrodes were easily fabricated using the apparatus shown in Fig. 4. The silver rod was connected to the positive (+) terminal of a 1.5 volt D cell, and a stainless steel strip was connected to the negative (-) terminal. The silver and the stainless steel were both immersed in a solution containing 100 g/L NaOH and 33 g/L Na₂S for a few minutes to form the brittle, grey-black film on the silver.

Once the electrodes were manufactured, no special precautions were taken in handling the electrodes. They could be stored in a dry condition and handled freely, yet achieve a stable and reproducible potential soon after immersion in a simulated white liquor. In some cases, the brittle film — determined by x-ray diffraction measurements to be Acanthite, Ag₂S — would be damaged, exposing the underlying silver rod. This had no detectable effect on the reference potential after a few minutes of immersion in hot white liquor.

The electrodes were prepared with an internal 5-40 UNC thread to facilitate mounting of the reference electrode to a standard Petrolite corrosion sensing probe as shown in Fig. 5. The electrodes were mounted in probes of this type by screwing the electrode against a hypalon or an ethylene-propylene gasket on a threaded rod extending from a glass-insulated feed-through.

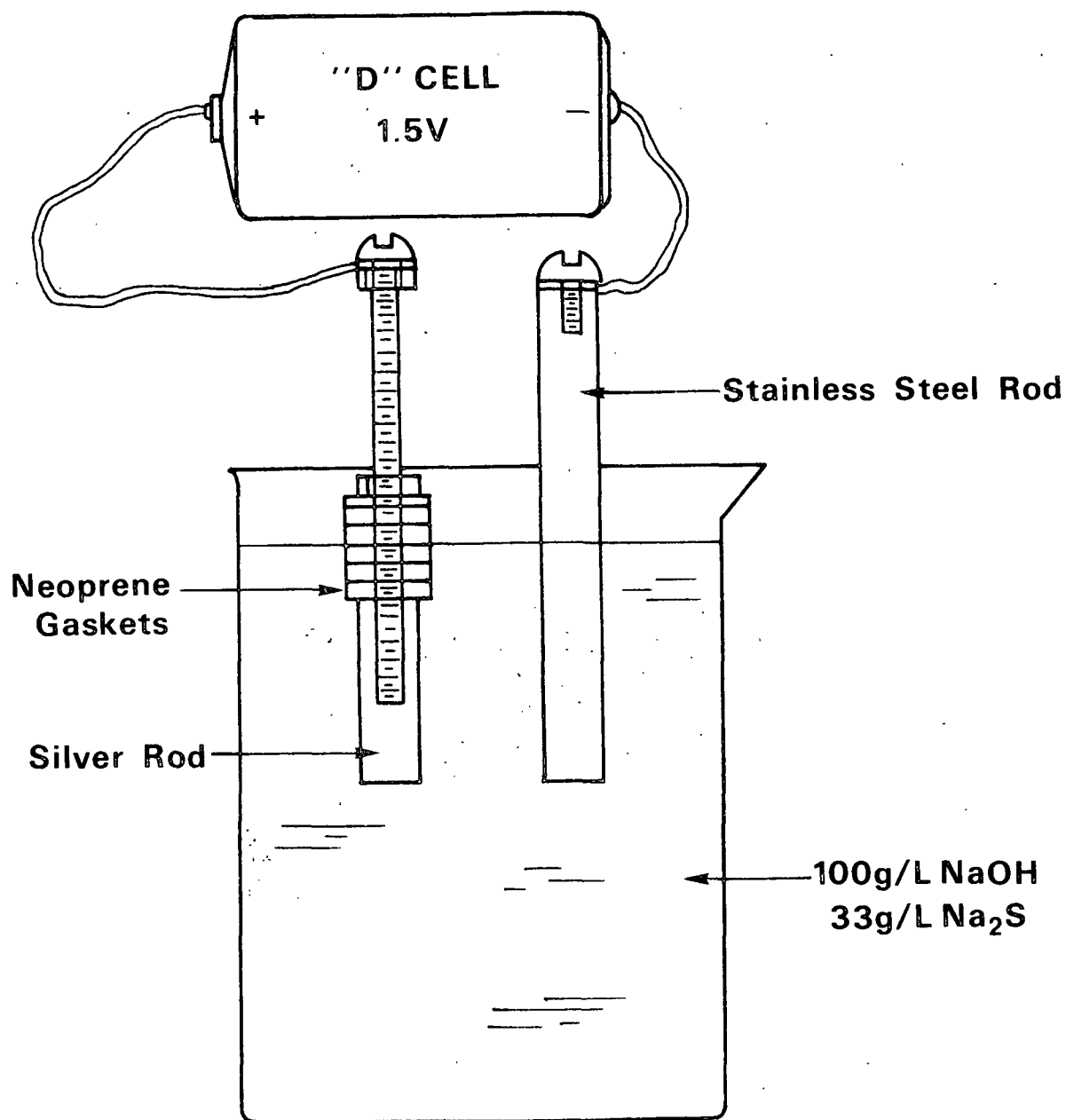


Figure 4. Apparatus for preparing Ag/Ag₂S reference electrodes from silver rods.

Experience also showed that a silver surface exposed to a hot alkaline sulfide solution will eventually achieve the reference potential of the Ag/Ag₂S electrode without the pretreatment described above. In fact, an Ag/AgCl electrode immersed in white liquor soon became a Ag/Ag₂S electrode by interaction with the sulfides in the liquor. Continued exposure of the Ag/Ag₂S electrode

resulted in a thickening of the Ag_2S film with concurrent loss of the underlying silver. Crude estimates of the rate of silver conversion to silver sulfide suggest that the 6 mm diameter probes would be substantially converted to Ag_2S in about a year of exposure to white liquor at 90°C . Whether this conversion will impair the stability of the rest potential is uncertain, but the complete formation of Ag_2S will surely damage the mechanical integrity of the electrode.

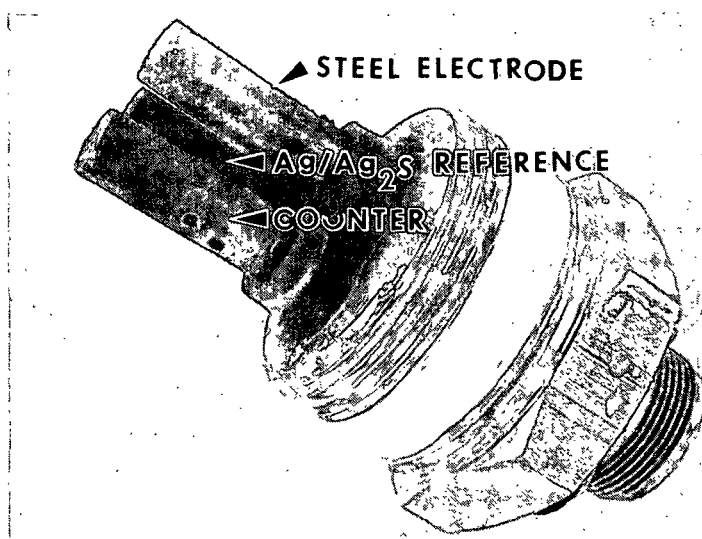


Figure 5. A standard Petrolite corrosion sensing probe with a SSS reference electrode mounted as shown.

ELECTRODE TESTING

The rest potential of the SSS electrode was measured vs. the saturated Calomel electrode (SCE) in a variety of simulated white liquors as well as several actual liquors extracted from various stages of the recausticizing and pulping processes. SSS potentials were determined vs. the SCE electrode by immersing the SSS electrode directly in the liquor while isolating the SCE

electrode from the sulfide environments by a double salt bridge. The SCE electrode was maintained at room temperature in this experiment. The experimental apparatus is shown schematically in Fig. 6. The double salt bridge consisted of asbestos wicks encapsulated in Teflon tubes and saturated with concentrated solutions of either NaOH or KCl.

Potential differences between the SSS and SCE electrodes were measured twice daily with a high impedance digital voltmeter over periods ranging from one week to one month. No attempt was made to correct the measurements for errors due to the thermal junction potentials or the liquid junction potentials developed with this configuration.

The SSS potential was measured in a variety of liquors at several different temperatures. Tests were conducted in simulated liquors with compositions in the range shown in Table I. Each chemical constituent likely to be found in white liquors was examined for its effect on the SSS potential at the limits shown in the table. In some cases, several intermediate concentrations of each of these species were also examined. Tests were conducted in simulated liquor temperatures of 70, 80, and 90°C as well as at room temperature to examine the effect of varying temperature on the rest potential of the SSS electrode. Not all of the tests were repeated for each solution at each temperature.

In addition to the tests in simulated liquors, additional tests were conducted in actual mill liquors to ascertain whether the actual liquors would behave differently from the simulated liquors. Some of these liquors were extracted from sites where substantial cooking of wood chips had occurred, so the liquors contained a significant concentration of typical organic by-products

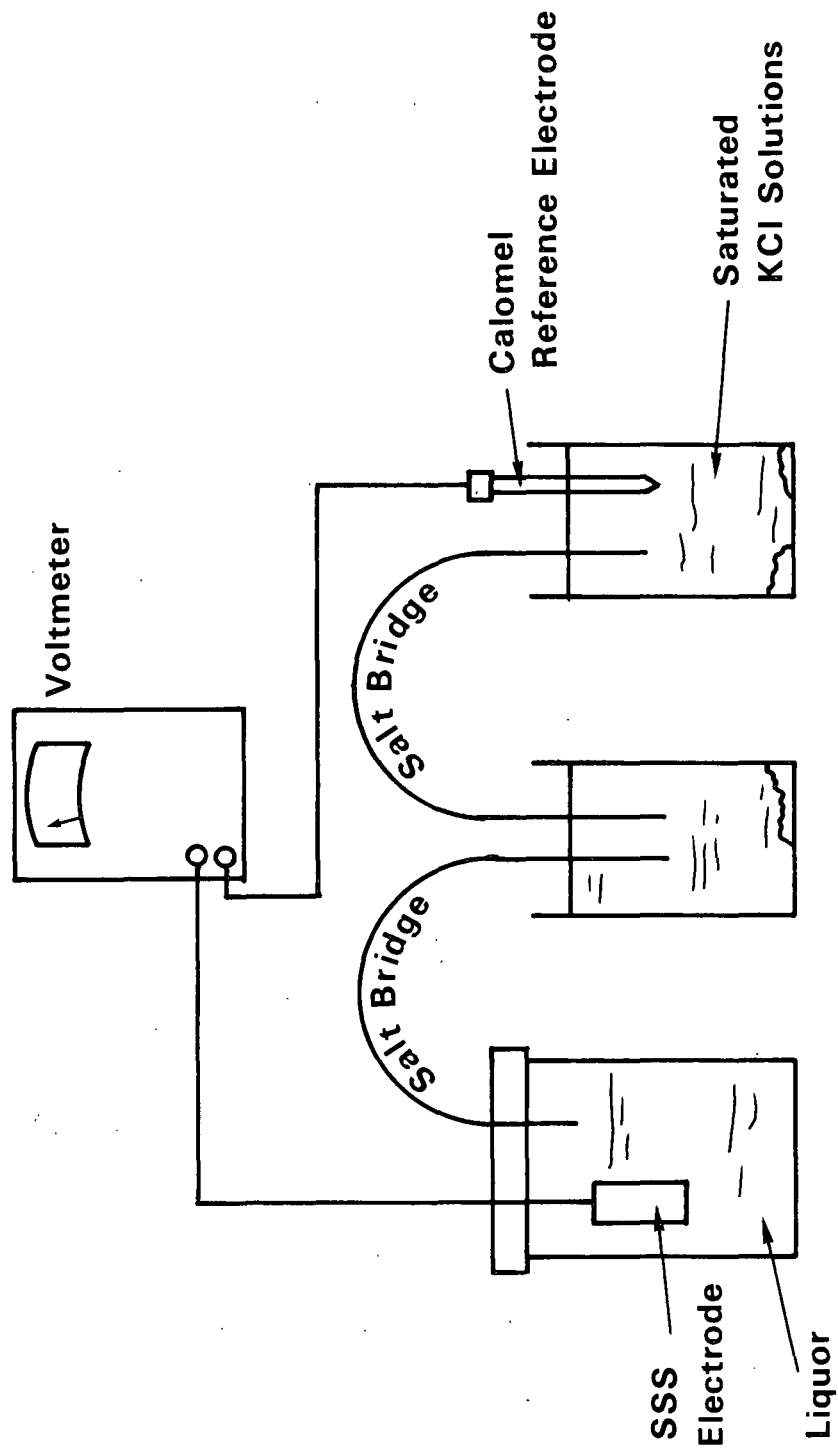


Figure 6. Schematic drawing of apparatus for measurement of the SSS potential vs. Calomel in white liquors.

of the early stages of kraft pulping. Tests in actual liquors were conducted at one temperature (90°C). The sources and compositions of the actual liquors tested are shown in Table II.

TABLE I
COMPOSITION RANGE FOR SIMULATED LIQUORS

| | Minimum, g/L | Maximum, g/L |
|---|-----------------|-----------------|
| NaOH | 85 | 100 |
| Na ₂ S | 6 | 42 |
| Na ₂ SO ₃ | 0 | 1 |
| Na ₂ SO ₄ | 0 | 10 |
| Na ₂ S ₂ O ₃ | 0 | 5 |
| Na ₂ S _x | 0 | 1.5 g/L as S° |
| NaCl | 0 | 5 |
| Na ₂ CO ₃ | 0 | 20 |

TABLE II
ACTUAL LIQUORS TAKEN FROM KAMYR DIGESTER LOOPS

| Mill No. | Site | NaOH, g/L | Na ₂ S, g/L | Na ₂ CO ₃ , g/L | NaCl, g/L | Na ₂ SO ₃ , g/L | Na ₂ SO ₄ , g/L | Na ₂ S ₂ O ₃ , g/L | S° |
|-------------|--------------------|--------------|---------------------------|--|--------------|--|--|--|----|
| 1 | Upper cook zone | 22 | 13 | 29 | 6 | 3 | 4 | 4 | 0 |
| 1 | Top circulation | 58 | 17 | 25 | 7 | 3 | 6 | 4 | 0 |
| 2 | Upper cook zone | 14 | 21 | 17 | 1 | 7 | 6 | 6 | 0 |
| 3 | Make-up liquor | 13 | 29 | 22 | 3 | 9 | 11 | 8 | 0 |
| 3 | Top circulation | 59 | 26 | 22 | 2 | 7 | 8 | 8 | 0 |

RESULTS

The SSS electrodes all assumed a stable, reproducible potential soon after immersion in the actual and simulated liquors examined in this study. Individual electrodes exhibited small potential fluctuations with time; the largest day-to-day fluctuation observed at any electrode was ± 10 mV, but most electrode potentials remained constant to within ± 5 mV over periods as long as one month. When several different electrodes were immersed at the same time in the same electrolyte, all of their rest potentials remained within ± 15 mV for exposures of up to one month.

The rest potential of the SSS electrode was slightly dependent on the concentration of the Na_2S in the host liquor, as shown in Fig. 7. The average potential measured during an exposure is shown in Fig. 7 as a data point, while the maximum and minimum potentials measured over time in multiple exposures is shown as a vertical scatter bar at the corresponding Na_2S concentration. The average SSS potential ranged from -865 mV SCE (-624 mV SHE) at 6 g/L Na_2S to -905 mV SCE (-664 mV SHE) at 42 g/L Na_2S . For the concentration of Na_2S typically encountered in kraft white liquors, 20 to 45 g/L Na_2S , the range of the average SSS potential is only -890 to -905 mV SCE.

None of the other species usually present in white liquors had an effect on the potential established by the Na_2S concentration, over the range of composition described in Table I. In particular, the SSS electrode potential was not affected by the deliberate addition of oxidizing species such as oxygen and polysulfide to the simulated white liquor, which establishes that the SSS potential is not a simple redox potential. None of the other inorganic species present in white liquor — NaOH , $\text{Na}_2\text{S}_2\text{O}_3$, Na_2SO_3 , Na_2SO_4 , Na_2CO_3 , NaCl — affected the SSS reference potential.

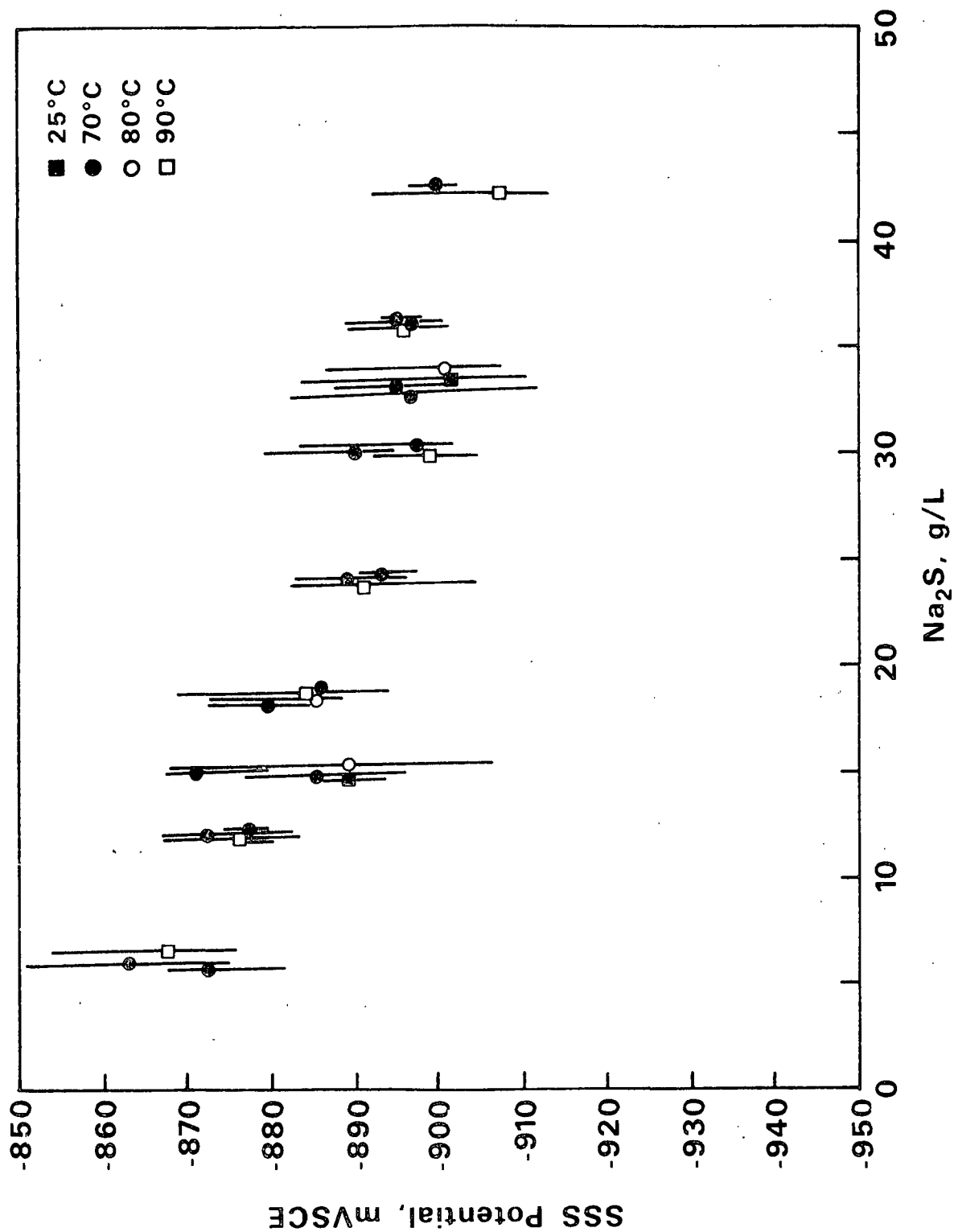


Figure 7. Dependence of SSS potential on Na₂S concentration at several temperatures. The data points represent average values during the exposure; the vertical bars represent the extent of fluctuation in potential for multiple exposures.

The temperature of the test solution also had relatively little effect on the SSS potential, as shown in Fig. 7. The limited data taken at room temperature are consistently lower in potential than the elevated temperature data, as would be expected from an examination of the Nernst equation. The differences in reference potential at 70, 80, and 90°C are insignificant.

Tests on actual mill liquors containing unknown quantities of decomposition products from low temperature wood pulping are shown in Fig. 8. The average SSS reference potential is at the upper limits of the scatter band for the average data shown for simulated liquors in Fig. 7, but there is considerable overlap in the scatter band for the data. However, the essential trend regarding the dependence of the SSS potential on $[\text{Na}_2\text{S}]$ concentration does not appear to be disturbed by the presence of the organic species in the liquors.

The rest potential of the SSS electrode was also insensitive to the exact details of preparation of the electrode and its treatment prior to immersion in the white liquor. Immersion of a dry electrode usually required a 20 minute incubation period before the electrode assumed the final potential. Immersion of a pure silver electrode without pretreatment usually required a longer time to adjust its potential to the observed reference potential, but even in this case the incubation time was short — on the order of one hour.

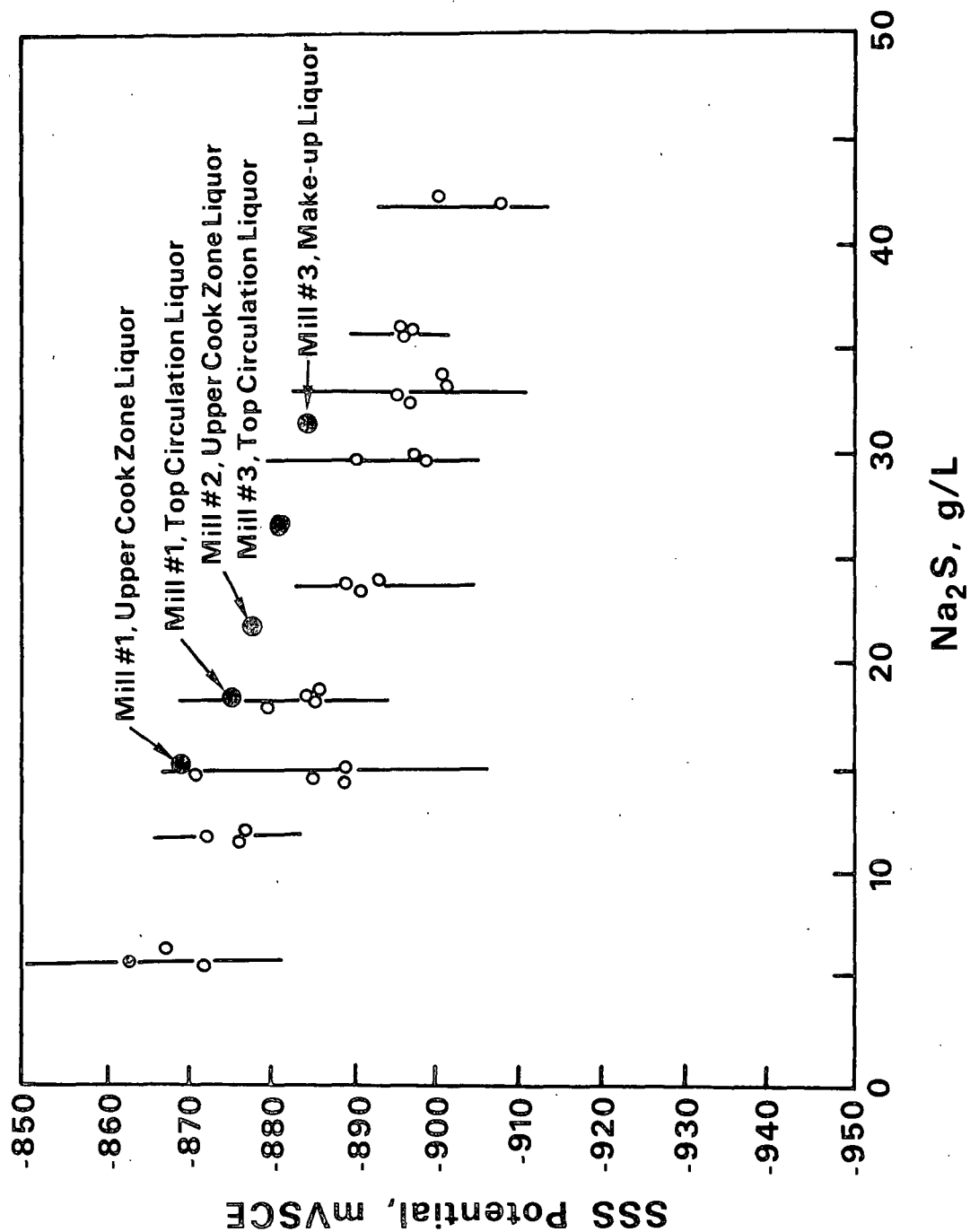
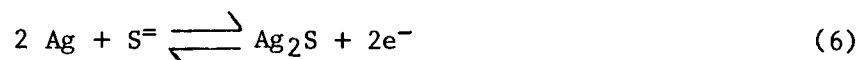


Figure 8. ESSS for actual mill liquors taken from several locations in continuous digester process streams from three different mills.

DISCUSSION

The silver/silver-sulfide electrode has long been identified as a candidate reference electrode, and there have been many investigations related to the SSS potential in alkaline sulfide media (2-7). Goates et al. (7) have concluded that the SSS potential is established by the reaction



which differs from the one-electron charge transfer event in the silver/silver-chloride electrode. However, Golding (6) has disputed the 2 electron event, finding instead that 1.67 electrons are transferred per event. Assuming that the reaction in Eq. (6) is accurate, the Nernst Equation for the reaction establishes the dependence of the SSS reference potential on the sulfide activity.

$$E_{\text{SSS}} (\text{vs. SHE}) = -0.7125 - \frac{RT}{nF} \ln a_{\text{S}^{=}} \quad (7)$$

where $a_{\text{S}^{=}}$ is the activity of the sulfide ion in the liquor. If the activity coefficient for the sulfide ion is assumed to be one, the activity of the $\text{S}^{=}$ ion can be replaced by the molar concentration of $\text{S}^{=}$. If we suppose that the pH of white liquor is approximately 14, the ratio of $[\text{HS}^{-}]$ to $[\text{S}^{=}]$ is approximately 10 [Pourbaix, Ref. (8)], so only one-tenth of the sulfide associated with a given concentration of Na_2S is available as the sulfide, $\text{S}^{=}$, at this pH. Thus, a solution containing 33 g/L Na_2S will only contain 0.038M $\text{S}^{=}$, and a solution containing 6 g/L Na_2S will only have 0.007M $\text{S}^{=}$. Inserting these concentrations for the activities in Eq. (7), and taking $\frac{RT}{2.3 nF} = 0.036$ at 90°C, we obtain reference potentials of -0.635 mV SHE (-0.874 V SCE) and -0.661 V SHE (0.901 V SCE), for $[\text{Na}_2\text{S}] = 6 \text{ g/L}$ and 33 g/L, respectively. These points and the curve corresponding to Eq. (7) are plotted in Fig. 9, together with the average data points

taken from the 90°C tests shown in Fig. 7. The agreement between the theoretical and measured values of E_{SSS} is reasonably good considering the assumptions involved, but the theoretical values (determined with the assumptions described above) lie approximately 10 mV below the average of the data shown in Fig. (7). The exact cause of the 10 mV discrepancy between the data and the theoretical equation is unknown, but may be the result of assumptions regarding the unit activity coefficient or, as suggested by Golding (6), an error in the use of $n = 2$ in the Nernst equation.

An empirical expression for E_{SSS} at 90°C based on the Na_2S concentration — rather than the $S^{=}$ concentration — is given as follows.

$$E_{SSS} \text{ (vs. SHE)} = -0.7125 - 0.039 \log \left(\frac{[Na_2S]}{858} \right) \quad (8)$$

where $[Na_2S]$ is the sodium sulfide concentration in g/L as chemical. This empirical curve is shown as the solid line of fit in Fig. 9.

The absence of an effect of nonsulfide species on the silver/silver electrode is not surprising if the exchange current density for reaction (7) establishes the rest potential. Other reactions that might occur on the electrode as a result of exposure to simulated and actual liquors do not affect the dominance of reaction (7) in establishing the rest potential. The organic species present in the actual liquors did raise the E_{SSS} slightly for a given $[Na_2S]$ concentration, but this shift in potential is only about 10 mV.

The effect of temperature on the E_{SSS} potential is also small, as would be expected from the Nernst equation for the Ag/Ag_2S reaction. The small effect of temperature is also evident in the results of a recent study (9) of the

electrochemical behavior of carbon steel in continuous digester liquors. In those tests, silver/silver-sulfide electrodes were calibrated in actual liquors (taken from make-up liquor pump suctions) at temperatures as high as 140°C. In those tests, the rest potential of the silver/silver sulfide electrode was found to be -872 ± 5 mV SCE at 140°C in liquors containing 27-34 g/L of Na_2S . The potential observed at 140°C is not remarkably different from the potential observed at 90°C in a simulated liquor of similar concentration.

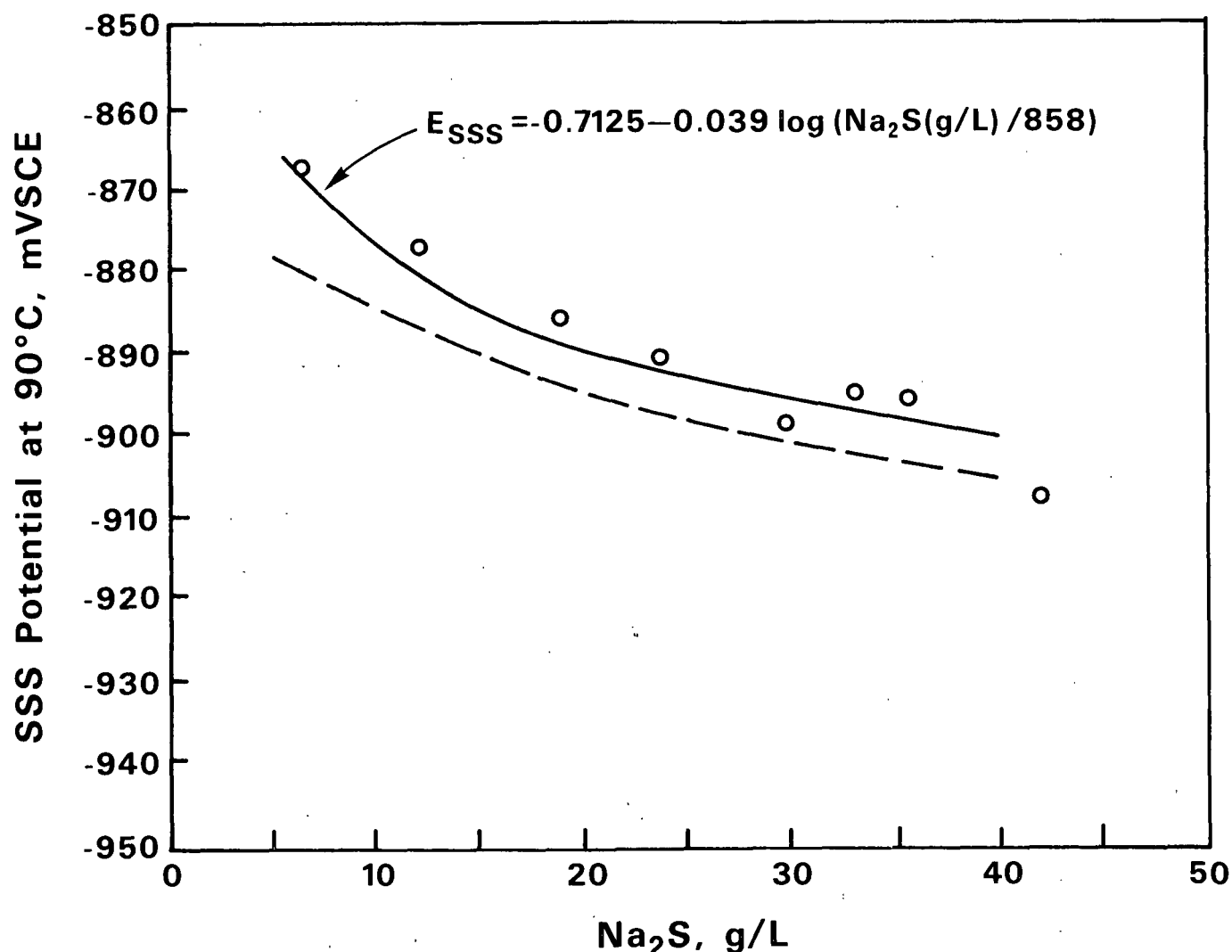


Figure 9. E_{SSS} vs. $[\text{Na}_2\text{S}]$ in simulated liquors at 90°C. The solid line of fit is given by $E_{\text{SSS}} (\text{vs. SHE}) = -0.7125 - 0.039 \log \left(\frac{\text{Na}_2\text{S}}{858} \right)$, where $[\text{Na}_2\text{S}]$ is in g/L. The dashed line is for Eq. (7).

One characteristic of reference electrodes not evident in the SSS electrode is nonpolarizability. Ideally, reference electrodes should pass significant currents though their electrode/electrode interfaces without large changes in potential — i.e., they should be nonpolarizable. Nonpolarizability insures that a constant reference potential will be maintained during use. Polarization tests on the Ag/Ag₂S electrode show that the cathodic polarization requires large current densities as desired, but anodic polarization occurs readily at a current density of only 0.4 mA/cm² (Fig. 10). This observation suggests that the greatest accuracy with SSS electrodes will be assured if voltage measurements are made with high impedance voltmeters or electrometers. Use of high impedance voltmeters will insure that small currents pass through the reference electrode surface when potential is being measured, thereby insuring that the reference potential does not shift due to polarization of the reference electrode.

Overall, the silver/silver-sulfide electrode appears to be ideal for use in electrochemical studies in white liquor. The electrode is easily fabricated and maintained, and is compatible with standard sensor probes. Furthermore, the electrode is durable and retains its accuracy after rough treatment or prolonged exposures in kraft liquors. The reference potential is stable enough for all but the most exacting electrochemical measurements in white liquor. The reference potential can easily be related to potentials of more familiar electrodes, such as the Calomel or Standard Hydrogen electrode. The precision of the SSS potential can be increased by measurement of the Na₂S concentration and corresponding calibration of the potential using Fig. 7 or Eq. (8). Based on an extensive investigation, it appears that the SSS reference electrode will provide a meaningful reference point for potential measurements in white liquor, without the need for the electrochemists training or paraphernalia.

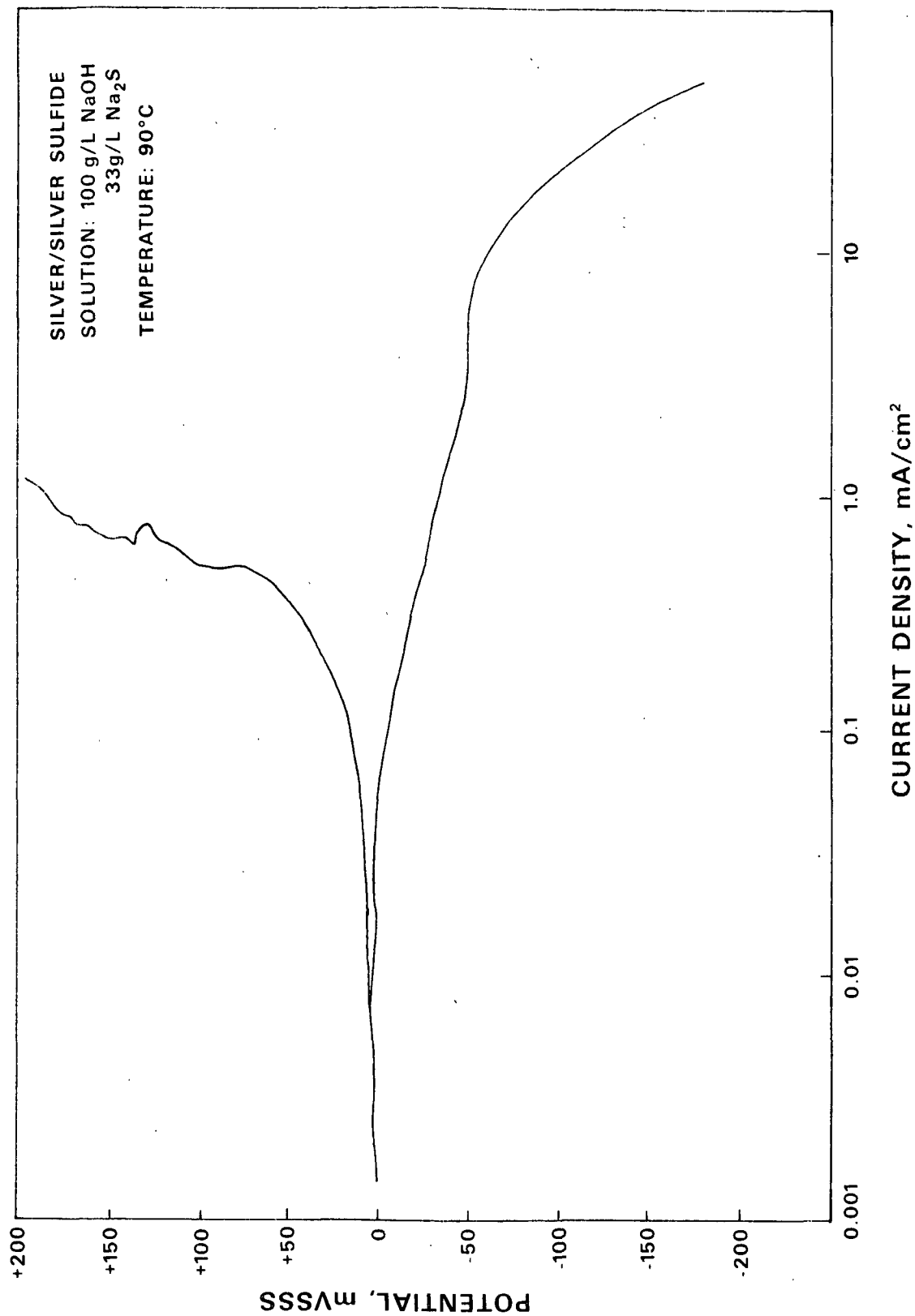


Figure 10. Anodic and cathodic polarization curves for the SSS electrode in a 90°C alkaline polysulfide solution.

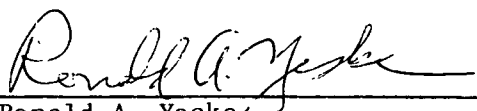
CONCLUSIONS

Tests have shown that the silver/silver-sulfide electrode has characteristics that make it ideal for use in electrochemical studies of corrosion in white liquor. The electrode is durable, easily fabricated, directly immersible, and can be mounted directly on sensor probes commonly used in field corrosion studies. The SSS reference potential is in the range -870 to -905 mV SCE, depending on the exact $[\text{Na}_2\text{S}]$ concentration in the liquor. The temperature sensitivity of the rest potential also appears to be slight over the range, 25 to 90°C. In contrast to other reference electrodes, the SSS electrode can be used in corrosion studies by mill site personnel without extensive training in electrochemical methods.

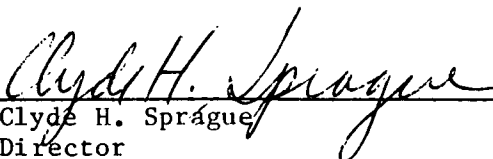
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